*भारतीय मानक*
*Indian Standard*

**IS 265 : 2021**

# हाइड्रोक्लारिक अम्ल — विशिष्टि

*( पाँचवा पुनरीक्षण )*

# Hydrochloric Acid — Specification

*( Fifth Revision )*

ICS 71.060.30

© BIS 2021

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

**March 2021** **Price Group 8**

Inorganic Chemicals Sectional Committee, CHD 01

## FOREWORD

This Indian Standard (Fifth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Hydrochloric acid is used for acidification (activation) of petroleum well, as chemical intermediate in ore reduction, food processing, pickling and metal cleaning, cleaning of membrane in the desalination plants, as alcohol denaturant and in boiler water plants.

This standard was first published in 1950 and subsequently revised in 1962, 1976, 1987 and 1993. In the fourth revision, a separate grade of hydrochloric acid for use in boiler water plant was incorporated. In this revision, Food grade of hydrochloric acid has been introduced. Road tankers have been included while screw capped stone ware bottles have been deleted in packing clause. Sulphates (as $H_2SO_4$) percent by mass, the factor has been corrected as 42.06 instead of 42.02. Further, silica dish has been included along with platinum dish for testing residue on ignition.

A correlation table for relative density and percent by mass of hydrochloric acid is given in Annex A for information.

Hydrochloric acid is a hazardous chemical and its safety aspects are covered in IS 6164 : 1971 ‘Code of safety for hydrochloric acid’.

The composition of the committee responsible for formulation of this standard is given at Annex P.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

# HYDROCHLORIC ACID — SPECIFICATION

## *( Fifth Revision )*

## 1 SCOPE

This standard prescribes requirements and methods of sampling and test for hydrochloric acid.

## 2 REFERENCES

The standards listed below contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

| *IS No.* | *Title* |
|---|---|
| 266 : 1993 | Sulphuric acid (*third revision*) |
| 1070 : 1992 | Reagent grade water (*third revision*) |
| 1260 (Part 1) : 1973 | Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*) |
| 2088 : 1983 | Methods for determination of arsenic (*second revision*) |
| 4905 : 2015/ISO 24153 : 2009 | Random sampling and randomization procedures (*first revision*) |

## 3 GRADES

There shall be following five grades of hydrochloric acid:

a) Technical grade (Tech),
b) Chemical pure grade (CP),
c) Food Grade (FG),
d) Analytical reagent grade (AR); and
e) Boiler water grade (BW).

## 4 REQUIREMENTS

### 4.1 Description

**4.1.1** *Technical Grade and Boiler Water Grade*

The material shall be a clear, colourless or light yellow liquid free from dirt and other visible impurities.

**4.1.2** *Food Grade*

The material shall be clear, colourless or light yellow liquid free from dirt and other visible impurities and organic materials.

The material produced only by synthesis of pure chlorine and hydrogen shall be suitable for food grade.

Hydrochloric acid obtained as a by-product from other chlor-organic processes, will not be suitable material for food grade unless it meets the requirement of total organic carbon as specified in the Table 1.

**4.1.3** *Chemically Pure and Analytical Reagent Grade*

The material shall be a colourless fuming liquid free from dirt and other visible impurities.

**4.2** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in col 8 of Table 1.

## 5 PACKING AND MARKING

### 5.1 Packing

**5.1.1** The material shall be packed in containers as agreed to between the purchaser and the supplier, and subject to the relevant provisions of Red Tariff No. 18 of 1960, issued by the Indian Railways Conference Association with any alterations or additions made thereafter. Bulk supply of hydrochloric acid is made in rubber lined MS road Tankers.

**5.1.1.1** When hydrochloric acid is supplied in glass carboys, polyethylene carboys or hard-rubber drums, the containers shall be fitted with leak-proof stoppers.

**5.1.1.2** Hydrochloric acid of analytical reagent grade shall be supplied in glass bottles or polyethylene containers or glass carboys fitted with tight-fitting HDPE or other suitable stoppers. The use of HDPE or other suitable caps over the stopper is recommended.

**5.1.1.3** The bottles and jars shall be packed in suitable pent-top packing cases. They shall be placed in an upright position on saw dust, straw, chalk or dry earth, and the empty surrounding space shall also be filled with the same material to prevent movement. Carboys shall be packed in suitable iron hampers or wooden crates, the interspace being sufficiently stuffed with whiting kiselguhr or other non-combustible absorbent material.

### 5.2 Marking

**5.2.1** The packages shall be suitably marked in red letters not less than 25 mm high showing the name of the acid; indication of the source of manufacture; and the grade and mass of the material. They shall prominently display the words 'CORROSSIVE, HANDLE WITH CARE'.

**5.2.2** In case of analytical reagent grade, the actual analysis of the material with respect to the characteristics laid down in co1 7 of Table l shall also appear on the label.

**5.2.3** The packages shall be labelled as shown in Fig. 15 of IS 1260 (Part 1).

### 5.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act,* 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 6 SAMPLING

Representative sample of the material shall be drawn and conformity of a lot to the standard determined as prescribed in Annex C.

## 7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemical and distilled water (*see* IS 1070) shall be used in tests.

> NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**Table 1 Requirements for Hydrochloric Acid**

( *Clauses* 4.1.2, 4.2 *and* 5.2.2 )

| **Sl No.** | **Characteristic** | **Requirement, Grade** | | | | | **Method of Test, Ref to Annex** |
|---|---|---|---|---|---|---|---|
| | | BW | Tech | Food1) | CP | AR | |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) |
| i) | Total acidity (as HCl), percent by mass, *Min* | 30.0 | 30.0 | 30.0 | 32.0 | 32.0 | B |
| ii) | Residue on ignition, percent by mass, *Max* | 0.01 | 0.1 | 0.01 | 0.01 | 0.0005<br>(5 ppm) | C |
| iii) | Sulphates (as $H_2SO_4$), percent by mass, *Max* | 0.1 | 0.1 | 0.01 | 0.01 | 0.0002<br>(2 ppm) | D |
| iv) | Iron (as Fe) percent by mass, *Max* | 0.002<br>(20 ppm) | 0.005<br>(50 ppm) | 0.001<br>(10 ppm) | 0.0001<br>(1 ppm) | 0.00004<br>(0.4 ppm) | E |
| v) | Free chlorine and bromine (as Cl), percent by mass, *Max* | 0.0005<br>(5 ppm) | 0.001<br>(10 ppm) | 0.001<br>(10 ppm) | 0.001<br>(10 ppm) | 0.000 2<br>(2 ppm) | F |
| vi) | Sulphites (as $SO_2$), percent by mass, *Max* | 0.05 | 0.05 | 0.03 | 0.04 | 0.0001<br>(1 ppm) | G |
| vii) | Heavy metals (as Pb) percent by mass, *Max* | 0.002<br>(20 ppm) | _ | 0.0005<br>(5 ppm) | 0.0005<br>(5 ppm) | 0.00008<br>(0.8 ppm) | H |
| viii) | Arsenic (as As), percent by mass, *Max* | 0.0005<br>(5 ppm) | _ | 0.000 05<br>(0.5 ppm) | 0.0002<br>(2 ppm) | 0.000 002<br>(0.02 ppm) | J |
| ix) | Ammonium compounds (as NH4), percent by mass, *Max* | _ | _ | _ | _ | 0.000 3<br>(3 ppm) | K |
| x) | Mercury (as Hg), ppm, *Max* | 3 | 3 | 0.01 | 0.03 | 0.0003 | L |
| xi) | Organic Carbon (as C), ppm, *Max* | – | – | 10 | – | – | M |

1) Virgin hydrochloric acid produced only from pure chlorine and hydrogen will be suitable for this grade, in order to exclude other organic and inorganic impurities associated in hydrochloric acid obtained from other chemical process as a by-product.

# ANNEX A

( *Foreword* )

## CORRELATION TABLE FOR RELATIVE DENSITY AND PERCENT BY MASS OF HYDROCHLORIC ACID

| Relative Density at 25 °C | Percent by Mass |
|---|---|
| (1) | (2) |
| 1.000 | 0.6 |
| 1.005 | 1.6 |
| 1.010 | 2.6 |
| 1.015 | 3.6 |
| 1.020 | 4.7 |
| 1.025 | 5.7 |
| 1.030 | 6.7 |
| 1.035 | 7.8 |
| 1.040 | 8.8 |
| 1.045 | 9.9 |
| 1.050 | 10.9 |
| 1.055 | 11.9 |
| 1.060 | 12.9 |
| 1.065 | 13.9 |
| 1.070 | 14.9 |
| 1.075 | 15.9 |
| 1.080 | 16.9 |
| 1.085 | 17.9 |
| 1.090 | 18.9 |
| 1.095 | 19.9 |
| 1.100 | 20.8 |
| 1 105 | 21.8 |
| 1.110 | 22.8 |
| 1.115 | 23.8 |
| 1.120 | 24.7 |
| 1.125 | 25.7 |
| 1.130 | 26.7 |
| 1.135 | 27.7 |
| 1.140 | 28.7 |
| 1.145 | 29.7 |
| 1.150 | 30.7 |
| 1.155 | 31.7 |
| 1.160 | 32.1 |
| 1.165 | 33.8 |
| 1.170 | 34.8 |
| 1.175 | 35.9 |
| 1.180 | 36.9 |
| 1.185 | 38.0 |
| 1.188 | 38.7 |

NOTE — This is the saturation value at 25 °C.

# ANNEX B

[ *Clause* 4.2, *Table* 1, *Sl No.* (i) ]

## DETERMINATION OF TOTAL ACIDITY

### B-1 OUTLINE OF THE METHOD

A known mass of the acid is carefully dissolved in water and titrated against standard alkali using methyl orange indicator.

### B-2 APPARATUS

**B-2.1 Lunge-Rey Pipette —** Lunge-rey pipette shall be of shape and dimensions as shown in Fig. 1. If this pipette is not available, a weighing bottle of a glass ampoule of the type shown in Fig. 2 may be used.

### B-3 REAGENTS

**B-3.1 Standard Sodium Hydroxide Solution***,* 1 N.

**B-3.2 Methyl Orange Indicator Solution***,* 0.05 g of methyl orange dissolved in 100 ml of water.

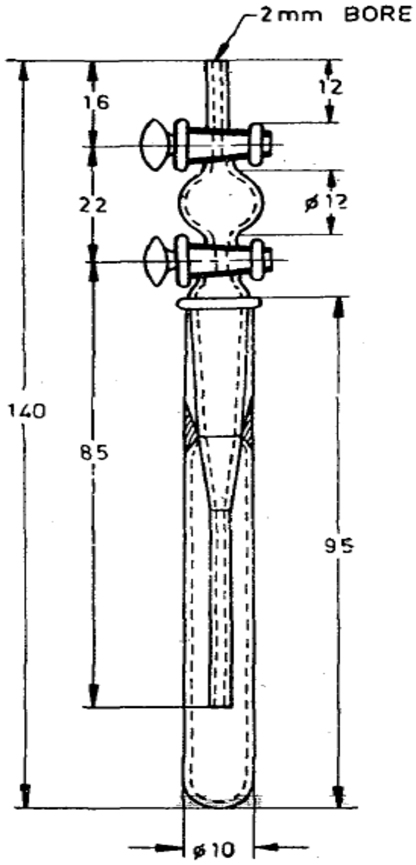
### B-4 PROCEDURE

**B-4.1** If Lunge-Rey pipette or an ordinary weighing bottle is used for weighing the sample, accurately weigh about 2 to 3 g of the material in it.

If a glass ampoule is used, take sufficient amount of sample in a beaker or flask. Slightly heat on flame the bulb of the glass ampoule, previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the beaker or flask containing the test sample and ensure that the bulb is filled up to about two-thirds of its volume during cooling (2 to 3 ml approximately). Withdraw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in an oxidizing flame without loss of glass. Remove the glass ampoule from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper. Weigh the ampoule to the nearest 0.1 mg and calculate by difference the mass of the test portion.

**B-4.2** If the sample was weighed in the Lunge-Rey pipette or weighing bottle, transfer the sample to a conical flask and dilute to 100 ml with water. If the sample was weighed in the ampoule, place it into the conical flask containing 200 ml of cold water. Stopper the flask and while cooling, shake to break the ampoule containing the test portion. Keep cooling and shaking until the vapors are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the conical flask. By means of a glass rod, mince the fragments of the ampoule and in particular the capillary which may have remained intact inspite of shaking. Withdraw the glass rod and wash it with water, collecting the washings in the conical flask.

**B-4.3** Add two drops of methyl orange indicator to the solution in the beaker or flask and titrate to the end point with standard sodium hydroxide solution.



All dimensions in millimetres

FIG. 1 LUNGE-REY PIPETTE

### B-5 CALCULATION

Total acidity (as HCl), percent by mass =

$$\frac{V \times N \times 3.646}{M}$$

where

$V$ = volume of standard sodium hydroxide solution used in titration, in ml;

$N$ = normality of standard sodium hydroxide solution; and

$M$ = mass of the sample taken for the test, in g.

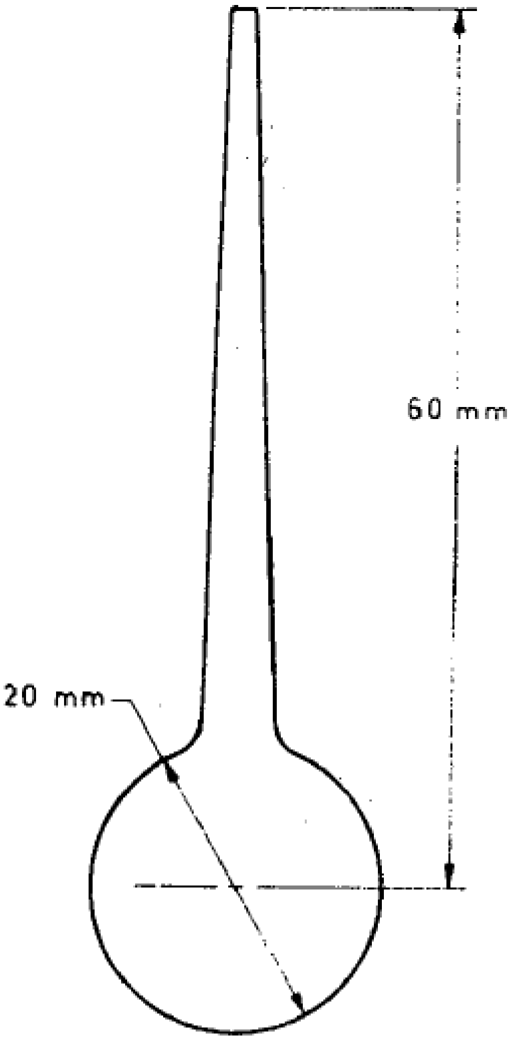


FIG. 2 SPHERICAL GLASS AMPOULE

# ANNEX C

[ *Table* 1, *Sl No*. (ii) ]

## DETERMINATION OF RESIDUE ON IGNITION

### C-1 OUTLINE OF THE METHOD

A known mass of the acid is evaporated, dried and the residue weighed.

### C-2 REAGENT

**C-2.1 Concentrated Sulphuric Acid —** *see* IS 266.

### C-3 PROCEDURE

In a platinum/silica dish previously ignited at 800 °C, cooled in a desiccator and weighed, weigh to the nearest 10 mg approximately 100 g of the test sample. Evaporate most of the acid (the final volume should amount to about 5 to 10 ml) by carefully heating the dish containing the test portion (on a sand bath, for example) in a fuming cupboard. Then allow to cool to room temperature. Add 1 ml of concentrated sulphuric acid and heat to dryness. Place the dish containing the residue in an electric furnace at 800 ± 5 °C and keep at this temperature for about 15 min. Remove the dish from the furnace, cool in a desiccator and weigh. Repeat the heating, cooling and weighing to constant mass.

### C-4 CALCULATION

$$\text{Residue on ignition, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

$M_1$ = mass of the residue, in g; and

$M_2$ = mass of the sample taken for the test, in g.

# ANNEX D

[ *Table* 1, *Sl No.* (iii) ]

## DETERMINATION OF SULPHATES

### D-1 OUTLINE THE METHOD

The sulphate in acid is precipitated as barium sulphate, dried and weighed.

### D-2 TECHNICAL AND BOILER WATER GRADES

**D-2.1 Reagents**

**D-2.1.1** *Sodium Carbonate* — Analytical reagent grade.

**D-2.1.2** *Barium Chloride Solution* — Approximately 10 percent (*m/v*).

**D-2.2 Procedure**

Weigh to the nearest 0.01 g about 10 g of the material ($M_1$) in a porcelain dish, add 0.5 g of sodium carbonate and evaporate to dryness. Moisten the residue with about 1.0 g of the material accurately weighed ($M_2$), add 200 ml of boiling water and filter, if necessary. Bring the contents to boil over a low flame and add slowly, with stirring, 5 ml of hot barium chloride solution. Boil the contents for 2 min and allow the precipitate to settle for 4 h. Filter the supernatant liquid through a tared sintered glass crucible (G No. 4) or a tared Gooch crucible and transfer the precipitate carefully into the crucible. Wash thoroughly with hot water till the washings are free from chloride. Heat the crucible at 105 to 110 °C to constant mass.

**D-2.3 Calculation**

$$\text{Sulphates (as } H_2SO_4\text{), percent by mass} = \frac{42.06\,M}{\left(M_1 + M_2\right)}$$

where

$M$ = mass of the precipitate, in g;

$M_1$ = mass of the sample taken for the test initially, in g; and

$M_2$ = mass of the sample added subsequently, in g.

### D-3 FOR CHEMICALLY PURE GRADE & FOOD GRADE

**D-3.1 Reagents**

*See* **D-2.1.**

**D-3.2 Procedure**

Follow the procedure, as given for technical grade in **D-2.2** using about 50.0 g of the material, weighed to the nearest 0.01 g, instead of 10 g.

**D-3.3 Calculation**

Calculate as in **D-2.3**.

## D-4 FOR ANALYTICAL REAGENT GRADE

### D-4.1 Reagents

**D-4.1.1** *Sodium Carbonate* — Analytical reagent grade.

**D-4.1.2** *Hydrochloric Acid* — Approximately 1 N, sulphate-free.

**D-4.1.3** *Barium Chloride Solution* — Approximately 10 percent (*m/v*).

**D-4.1.4** *Standard Sulphate Solution*

Dissolve 0.178 g of potassium sulphate in water and make up the volume to 1 000 ml. Dilute 100 ml of this solution in a graduated flask to 1000 ml. One millilitre of diluted solution contains 0.01 mg of sulphate (as $H_2SO_4$).

### D-4.2 Procedure

Add 20 mg of sodium carbonate to 20.00 g of the material and evaporate to dryness. Take up the residue with 5 ml of water and 0.5 ml of hydrochloric acid. Filter, wash with water to make up to 10 ml and add 1 ml of barium chloride solution. Carry out a control test in the same manner using 4 ml of standard sulphate solution in place of the material. Stir the two solutions and compare the turbidity produced.

**D-4.2.1** The limit prescribed in Table 1 shall be taken as not having been exceeded, if the turbidity produced with the material is not greater than that produced in the control test.

# ANNEX E

[ *Table* 1, *Sl No.* (iv) ]

## DETEMINATION OF IRON

## E-1 GENERAL

Two methods are prescribed. Method A shall be the referee method and Method B the alternative method.

## E-2 METHOD A (BIPYRIDYL METHOD)

### E-2.1 Outline of the Method

Iron is separated as coloured complex with 2, 2−bipyridyl and estimated spectrophotometrically.

### E-2.2 Apparatus

**E-2.2.1** *Photometer* — Any spectrophotometer suitable for measurement at a wavelength of about 522 *nm,* or a photoelectric absorptiometer.

### E-2.3 Reagents

**E-2.3.1** *Dilute Hydrochloric Acid* — 1 N approximately.

**E-2.3.2** *Hydroxylammonium Chloride Solution* — Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

**E-2.3.3** *2, 2 — Bipyridyl Solution*

Dissolve 1 g of 2, 2-bipyridyl in 10 ml of dilute hydrochloric acid (1 N) and dilute to 100 ml with water.

**E-2.3.4** *Standard Iron Solution A*

Dissolve 0.702 2 g of ferrous ammonium sulphate [$FeSO_4\,(NH_4)_2SO_4.6H_2O$] in water in a 1 000 ml volumetric flask, add 4 ml of concentrated sulphuric acid and make up to the mark with water. One millilitre of this solution contains 0.1 mg of iron (as Fe).

**E-2.3.5** *Standard Iron Solution B*

Take 100 ml of the standard iron solution A (*see* **E-2.3.4**) and dilute to 1 000 ml with water in a 1 000 ml volumetric flask. This dilute solution should be prepared fresh. One milliliter of this solution contains 0.01 mg of iron (as Fe).

**E-2.3.6** *Ammonium Acetate Solution*

Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

### E-2.4 Procedure

**E-2.4.1** Weigh accurately about 50 g of the sample in a platinum or silica dish (100 ml capacity), place on a boiling water bath and carefully evaporate to dryness. Cool, take up with 2 ml of dilute hydrochloric acid and 25 ml of water, and heat to facilitate dissolution. Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and filter, if necessary. Transfer an aliquot of the sample solution containing between 50 and 500 µg of iron to a 100 ml one-mark volumetric flask. Dilute to approximately 50 ml, if necessary, and then add successively 2 ml of dilute hydrochloric acid solution, 2 ml of hydroxyl ammonium chloride solution and, after 5 min, 5 ml of ammonium acetate solution and 1 ml 2, 2-bipyridyl solution. Dilute to the mark, mix and wait for 10 min. Carry out the measurement on the spectrophotometer at a wavelength of about 522 nm, adjusting the instrument to zero optical density using as reference the blank test.

**E-2.4.2** *Blank Test*

At the same time, carry a blank test using the same procedure and quantities of all reagents employed in the test.

**E-2.4.3** Prepare a calibration curve by taking the quantities of standard iron solution indicated below in a series of eleven 100 ml volumetric flasks:

| *Volume of Standard Iron Solution* (*B*) ml | *Corresponding Mass of Iron* (Fe) µg |
|---|---|
| 0 (Compensation solution) | 0 |
| 5.0 | 50 |
| 10.0 | 100 |
| 15.0 | 150 |
| 20.0 | 200 |
| 75.0 | 250 |
| 30.0 | 350 |
| 40.0 | 400 |
| 45.0 | 450 |
| 50.0 | 500 |

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of dilute hydrochloric acid solution, 2 ml of hydroxylammonium chloride solution and after 5 min, 5 ml of ammonium acetate solution and 1 ml of 2,2-bipyridyl solution. Dilute to the mark, mix thoroughly and wait for 10 min.

**E-2.4.4** Carry out the photometric measurement as in **E-2.4.1**, adjusting the instrument to zero optical density using as reference the compensation solution. Prepare a calibration curve having, for example, the iron content in micrograms per 100 ml of the standard matching solution as abscissa and the corresponding values of absorbance as ordinate.

**E-2.5 Calculation**

$$\text{Iron content (as Fe), percent by mass} = \frac{m \times 100 \times 100}{V \times M}$$

where

$m$ = mass of iron determined in the aliquot of sample solution, in g;

$V$ = volume of the sample taken for colour reaction, in ml; and

$M$ = mass of the material taken for test, in g.

## E-3 METHOD 2 (THIOCYANATE METHOD)

**E-3.1 Outline of the Method**

Iron is extracted with butanolic potassium thiocyanate and estimated by comparing colorimetrically.

**E-3.2 Apparatus**

**E-3.2.1** *Nessler Cylinders* — 50 ml capacity.

**E-3.3 Reagents**

**E-3.3.1** *Ammonium Persulphate*

**E-3.3.2** *Butanolic Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously till the solution is clear.

**E-3.3.3** *Standard Iron Solution B*

Take 100 ml of standard iron solution A (*see* **B-5.3.4**), add dilute potassium permanganate solution (0.2 percent, *m/v* ) until a slight pink colour persists after stirring and dilute to the mark in a 1000 ml volumetric flask. This dilute solution should be prepared afresh. One millilitre of this solution contains 0.01 mg of iron (as Fe).

**E-3.4 Procedure**

**E-3.4.1** *For Technical and Boiler Water Grades*

Dilute l.00 g of the technical grade material (20.00 g in case of boiler water grade) to 100 ml with water. Transfer 10 ml of this solution to a Nessler cylinder, add about 30 mg of ammonium persulphate, boil for a few min, cool and add 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for about 30 s and allow the layers to separate. Carry out a control test in dilute hydrochloric acid and 2 ml of the standard iron solution B in place of the material. Compare the intensity of the colour produced in the butanol layers in the two cylinders.

**E-3.4.2** *For Chemically Pure Grade*

Weigh 50.0 g of the material and evaporate it almost to dryness. Dilute it to 30 ml, add about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for about 30 s and allow layers to separate. Carry out a control test in another Nessler cylinder using 5 ml of standard iron solution B. Compare the intensity of the colour produced in the butanol layers in the two cylinders.

**E-3.4.3** *For Analytical Reagent Grade*

Carry out the test as given for chemically pure grade in **E-3.4.2**, using 2 ml of the standard iron solution B for the control test.

**E-3.5** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

# ANNEX F

[ *Table* 1, *Sl No.* (v) ]

## DETERMINATION OF FREE CHLORINE AND BROMINE

### F-1 OUTLINE OF THE METHOD

The material is shaken with potassium iodide starch solution and estimated iodimetrically.

### F-2 TECHNICAL GRADE

### F-2.1 Reagents

**F-2.1.1** *Potassium Iodide*, *crystals.*

**F-2.1.2** *Starch Solution* — Make a paste of 0.5 g of starch with 2.5 ml of water. Pour the paste in small quantities into 200 ml of water. Boil for 15 min with stirring. Preserve in small containers previously sterilized in boiling water for 2 h.

**F-2.1.3** *Potassium Iodide Solution*, 10 percent.

**F-2.1.4** *Standard Sodium Thiosulphate Solution*, 0.1 N.

### F-2.2 Procedure

**F-2.2.1** *Preliminary Test*

Place about 20 ml of the test sample in a l00 ml conical flask, add 50 ml of water, one crystal of potassium iodide, 0.5 ml of starch solution and stir. If a blue colour indicating iodine liberation appears, follow the procedure prescribed in **F-2.2.2** for determination of free chlorine and bromine. If no colour appears, proceed for the determination of sulphites as in Annex G.

**F-2.2.2** Fill a ground glass stoppered weighing bottle with test sample and take a portion of approximately 50 g, weighing by difference to the nearest 10 mg. Transfer the test portion to a conical flask fitted with ground glass stopper of capacity 500 ml containing 100 ml of water. Stopper the flask and cool. Add to the conical flask, 10.0 ml of potassium iodide solution, stopper and flask and shake. Allow to stand for 2 min and then add 1 ml of starch solution. Allow to stand for 2 min and then titrate with standard sodium thiosulphate solution until the blue colour disappears.

**F-2.2.3** Since iron may interfere in the determination, carry out a blank determination using 100 ml of water, and adding to it a water-soluble ferric iron salt equivalent to that contained in 50 g of the sample. Add other reagents as added to the sample. Subtract the volume of sodium thiosulphate solution required for titration from the volume required with sample (*see* **F-2.2.2** ).

### F-2.3 Calculation

Free chlorine and bromine (as $Cl_2$), percent by mass =

$$\frac{V \times m \times 100}{M}$$

where

$V$ = volume of 0.1 sodium thiosulphate solution used in titration (corrected for the blank), in ml;

$m$ = mass of chlorine corresponding to 1 ml 0.1 sodium thiosulphate solution (the oretical value, 1 ml = 0.00355 g of $Cl_2$), in g; and

$M$ = mass of the test portion, in g.

### F-3 FOR BOILER WATER, CHEMICALLY PURE AND ANALYTICAL REAGENT GRADES

### F-3.1 Apparatus

**F-3.1.1** *Microburette*

### F-3.2 Reagents

**F-3.2.1** *Cadmium Iodide Solution* — Dissolve 9.2 g of cadmium iodide in 100 ml of water.

**F-3.2.2** *Starch Solution* — *See* **F-2.1.2**.

**F-3.2.3** *Standard Sodium Thiosulphate Solutions* — 0.05 N and 0.005 N.

### F-3.3 Procedure

Dilute 4 ml of the material with 20 ml of freshly boiled and cooled water. Add 1 ml of cadmium iodide solution and 1 ml of starch solution, and allow to stand in the dark for 10 min.

**F-3.4** The limit prescribed in Table 1 shall be taken as not having been exceeded, if any blue colour produced on treating the material as in **F-3.3** is discharged by the addition of not more than 0.05 ml of 0.05 N sodium thiosulphate solution for chemically pure grade, not more than 0.125 ml of 0.005 N sodium thiosulphate solution in the case of boiler water grade and not more than 0.05 ml of 0.005 N sodium thiosulphate solution in the case of analytical reagent grade.

# ANNEX G

[ *Table* 1, *Sl No.* (vi) ]

## DETERMINATION OF SULPHITES

### G-1 OUTLINE OF THE METHOD

The material is shaken with standard iodine solution and estimated iodimetrically.

### G-2 FOR BOILER WATER, TECHNICAL AND CHEMICALLY PURE GRADES

**G-2.1 Reagents**

**G-2.1.1** *Standard Iodine Solution* — 0.1 N, freshly standardized.

**G-2.1.2** *Standard Sodium Thiosulphate Solution* — 0.1 N, freshly standardized.

**G-2.1.3** *Starch solution* — *See* **F-2.1.2**.

**G-2.2 Procedure**

Introduce successively into a 500 ml glass stoppered conical flask 100 ml of freshly boiled water and 10 ml of standard iodine solution. Add, while cooling, about 50 g of the material weighed to ±0.01 g. Stopper the flask, mix and after 2 min titrate the excess iodine with standard sodium thiosulphate solution, adding 5 ml of starch solution towards the end of the titration.

**G-2.1.3 Calculation**

Sulphites (as $SO_2$), percent = $\frac{0.3203\,(10 - V)}{M}$

where

$V$ = volume of standard sodium thiosulphate solution used in titration, in ml; and

$M$ = mass of the material taken for the test, in g.

### G-3 ANALYTICAL REAGENT GRADE

**G-3.1 Reagents**

**G-3.1.1** *Potassium Iodine Solution* — 10 percent (*m/v*).

**G-3.1.2** *Standard Iodine Solution* — 0.01 N.

**G-3.1.3** *Starch Solution* — *See* **F-2.1.2.**

**G-3.2 Procedure**

To 400 ml of freshly boiled and, cooled water, add 1.5 ml of potassium iodide solution, 5 ml of hydrochloric acid and 2 ml of starch solution and titrate with 0.01 N iodine solution to a faint permanent blue colour. Add 100 g of the sample and titrate with 0.01 N iodine solution to the same end point. The limit prescribed in Table 1 shall be taken as not having been exceeded if not more than 0.25 ml of 0.01 N iodine is required for the titration.

# ANNEX H

[ *Table* 1, *Sl No.* (vii) ]

## DETERMINATION OF HEAVY METALS

### H-1 OUTLINE OF THE METHOD

The presence of heavy metals is indicated from the turbidity formed when hydrogen sulphide is passed through the material.

### H-2 APPARATUS

**H-2.1** *Nessler Cylinders* — 50 ml capacity.

### H-3 REAGENTS

**H-3.1 Dilute Hydrochloric Acid —** 1 : 1 (*v/v*).

**H-3.2 Dilute Ammonia Solution —** Dilute 280 ml of strong ammonia solution to 1000 ml with water.

**H-3.3 Dilute Acetic Acid** — Dilute 285 ml of acetic acid to 1000 ml with water.

**H-3.4 Standard Iron Solution** — *See* **E-2.3.4.**

**H-3.5 Standard Lead Solution** — Dissolve 0.160 g of lead nitrate in water, add 1 ml of concentrated nitric acid and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 100 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

**H-3.6 Hydrogen Sulphide Gas**

### H-4 PROCEDURE

Accurately weigh 2.5 g of the material in the case of boiler water grade, 10 g in the case of chemically pure grade and 50 g in the case of analytical reagent grade in a platinum or silica crucible (100 ml capacity) and evaporate to dryness on a steam bath. Dissolve the residue by heating to boiling with a mixture of 3 ml dilute hydrochloric acid and 10 ml of water. Cool, dilute with 20 ml of water and transfer to a Nessler cylinder. Add 10 ml of dilute ammonia solution and standard iron solution equivalent to the amount of iron present in 50 g of the sample as determined in accordance with Annex E, and make the volume to 50 ml with water. In another Nessler cylinder, take 5 or 4 ml of standard lead solution according as the material is of boiler water grade, chemically pure grade or analytical reagent grade, for control test. Add 3 ml of dilute acetic acid solution, the same amount of standard iron solution as taken in the case of sample, and 10 ml of dilute ammonia solution. Make the volume to 50 ml. Pass hydrogen sulphide in both the Nessler cylinders for a few seconds.

**H-5** The limit prescribed in Table 1 shall be taken as not having been exceeded, if the turbidity produced in the test with the sample is not greater than that produced in the control test.

## ANNEX J

[ *Table* 1, *Sl No.* (viii) ]

## DETERMINATION OF ARSENIC

### J-1 OUTLINE OF THE METHOD

Arsenic is extracted with silver diethyl dithiocarbamate as a red complex and measured spectrophotometrically.

### J-2 PROCEDURE

Take a suitable quantity of the material so that the test solution contains 1 to 10 µg of arsenic in a final volume of 5.0 ± 0.5 ml. Determine arsenic by silver diethyl dithiocarbamate method as prescribed in IS 2088.

## ANNEX K

[ *Table* 1, *Sl No.* (ix) ]

## DETERMINATION OF AMMONIUM COMPOUNDS

### K-1 OUTLINE OF THE METHOD

The material is made slightly alkaline and shaken well with Nessler's reagent and the colour developed is compared with a standard.

### K-2 REAGENTS

**K-2.1 Sodium Hydroxide Solution** — 5 N, ammonia-free.

**K-2.2 Standard Ammonium Solution** — Dissolve 2.97 g of ammonium chloride in water and dilute to 1000 ml. One millilitre of this solution contains 1 mg of ammonium. Dilute 1 ml of this solution to 100 ml with water immediately before use to get 0.01 mg of ammonium per ml .

**K-2.3 Nessler's Reagent** — Dissolve 35 g of potassium iodide and 12.5 g of mercuric chloride in 800 ml of water and add a saturated solution of mercuric chloride until a slight permanent precipitate is produced. Then add 120 g of sodium hydroxide and dissolve. Add a little more mercuric chloride solution and sufficient water to make up to 1000 ml. Shake the solution occasionally for several days. Allow to settle and decant the clear liquid.

### K-3 PROCEDURE

Dilute 2.8 ml (3.3 g) of the material to about 20 ml with water and add sodium hydroxide solution until alkaline to litmus paper (about 7 ml is required). Dilute to 50 ml with water. Prepare a standard by diluting 1 ml of standard ammonium solution to 50 ml with water. To both solutions, add 2 ml of Nessler's reagents and compare after 1 min.

**K-4** The limit prescribed in Table 1 shall be taken as not having been exceeded if the yellow colouration produced, if any, with the test material is not deeper than that of the standard.

# ANNEX L

[ *Table* 1, *Sl No.* (x) ]

## DETERMINATION OF MERCURY

### L-1 GENERAL

Two methods have been specified, namely, dithizone method and mercury analyzer method. In case of dispute, the mercury analyzer method shall be taken as the referee method.

### L-2 DITHIZONE METHOD

### L-2.1 Outline of the Method

Mercuric mercury forms a complex with dithizone in the form of orange/red colour depending upon its concentration.

### L-2.2 Reagents

**L-2.2.1** *Dithizone* — Concentrated stock solution (25 to 30 mg) in 500 ml of chloroform; 25 mg of dithizone is dissolved in 100 ml of chloroform and extracted with 250 ml of 2 N ammonia. Most of the dithizone will pass into the aqueous layer and change colour from green to orange. The oxidation products and contaminants are retained in the chloroform layer which is separated and discarded. 500 ml of chloroform are added to the aqueous layer in the separating funnel and the solution is mixed with 6 ml of glacial acetic acid and gradually 1 : 1 sulphuric acid is added till the orange phase loses its colour. The solution is shaken well. When precipitated dithizone dissolves in the chloroform layer, this is separated and filtered. The volume is made up to 500 ml and the solution is stored in an amber coloured bottle. Stability of the prepared reagent is 10 days.

**L-2.2.2** *Diluted Dithizone* — Dilute 2 ml of the concentrated dithizone solution to 100 ml with chloroform (1 ml = 1 microgram of Hg). Stability of this reagent is 1 day.

**L-2.2.3** *EDTA* (*5 percent*) — Dissolve 5 g of disodium salt of EDTA in 100 ml of water.

**L-2.2.4** *Potassium Thiocyanate Solution* — 10 percent ( *m/v* ).

**L-2.2.5** *Sodium Acetate Buffer* (*p*H 5) — Weigh 56 g of sodium acetate and dissolve in water. Add 24 ml of glacial acetic acid. Make up to 100 ml with water.

**L-2.2.6** *Sulphuric Acid* — 10 percent.

**L-2.2.7** *Standard mercury solution* (*1 ml = 1 microgram of mercury*) — Dissolve 0.135 4 g of pure mercuric chloride in water and make up to 1000 ml. 10 ml of this solution is further diluted to 1000 ml. 1 ml of this solution contains 1 microgram of mercury.

**L-2.2.8** *Ammonia* Solution – 1 : 4.

**L-2.2.9** *Potassium Permanganate Solution* (*4 Percent*) — Dissolve 4 g of potassium permanganate in 100 ml of water.

**L-2.2.10** *Hydroxylamine Hydrochloride* (*2 Percent*) — Dissolve 2 g of hydroxylamine hydrochloride in 100 ml of water.

**L-2.2.11** *Chloroform*

**L-2.2.12** *Glacial Acetic Acid*

### L-2.3 Procedure

Neutralize 10 ml of the sample with 1 : 4 ammonia solution in a 250 ml beaker. Add 10 ml of 10 percent sulphuric acid followed by 1 ml of 4 percent potassium permanganate solution. Digest the same for 30 min, cool and neutralize the excess potassium permanganate with 2 percent hydroxylammonium chloride solution. Transfer the solution in a 500 ml separating funnel. Dilute to 100 ml. Now add 10 ml of sodium acetate buffer, 10 ml of 5 percent EDTA solution and 2 ml of 10 percent potassium thiocyanate solution. The solution is now saturated with 5 ml chloroform. Drain off the organic layer. Add 5 ml of glacial acetic acid and mix the solution thoroughly. Now add diluted dithizone solution (1 ml = 1 microgram of Hg ) in small quantities and extract separately till the dithizone solution remains green in colour. Collect the dithizone extract and not the volume. By known quantities of standard mercury solution, running a blank and following the same method of extraction standardized the dithizone solution.

### L-2.4 Calculation

$$\text{Mercury (as Hg), ppm} = \frac{V \times N}{10 \times d}$$

where

$V$ = volume of dithizone solution, in ml;

$N$ = strength of dithizone solution in pg, and

$d$ = relative density of hydrochloric acid.

### L-3 MERCURY ANALYZER METHOD

### L-3.1 Outline of the Method

Mercury analyzer works on the principle of cold vapour atomic absorption spectrometry technique. Mercury ions are reduced to elemental state by stannous chloride and the solution is stirred vigorously so that an equilibrium is achieved between the mercury in solution and air phase. The vapour is then purged into the absorption cell which is located in the light path of the spectrometer.

As the cold vapour absorption technique of mercury is based on absorption of UV radiation by mercury atoms, all substances which absorb UV radiation will cause the positive error. Vapours of organic compounds like alcohols, ketones, esters, and acids and water also absorb UV radiation. These can be avoided with suitable trap provided along with the instrument.

### L-3.2 Mercury in the Chemically Pure Grade

**L-3.2.1** *Reagents*

**L-3.2.1.1** *Nitric acid* — 10 percent (*v/v*).

**L-3.2.1.2** *Stannous chloride* — 20 g in 10 percent hyhrochloric acid (*m/v*).

**L-3.2.1.3** *Potassium permanganate* — 1 percent (*m/v*).

**L-3.2.1.4** *Hydroxylamine hydrochloride* — 10 percent (*m/v*).

**L-3.2.1.5** *Nitric acid* — 5 percent (*m/v*).

**L-3.2.1.6** *Potassium dichromate solution* — 1 percent (*m/v*).

**L-3.2.2** Apparatus

**L-3.2.2.1** *Mercury analyzer* EC MA/5 800 A or equivalent.

**L-3.2.3** Procedure

**L-3.2.3.1** *Preparation of sample solution*

Take 50 ml of the sample in a 100 ml standard volumetric flask. Add potassium permanganate solution drop-wise till the purple colour remains, add 2 to 3 drops of concentrated nitric acid. Keep it for 5 to 10 min after shaking flask. Decolourize the solution by adding hydroxylamine hydrochloride solution dropwise. Make up the volume to 100 ml with mercury free water.

Similarly, make the blank solution (B. S.) by using 50 ml mercury free water instead of hydrochloric acid and using the same quantity of reagents.

**L-3.2.3.2** *Preparation of standard graph*

a) *Preparation of stock solution* — Dissolve 0.135 4 g of mercuric chloride AR or GR grade in 25 ml of 2 percent of nitric acid (*v/v*). Add 2 ml of 1 percent (*m/v*) potassium dichromate solution of and make up to 100 ml with 5 percent nitric acid (*v/v*).

   1 ml of this stock solution = 1 mg of Hg.

b) *Preparation of standard solution* — By successive dilution of the above stock solution, prepare the standard solution of 1 ml = 100 nanogram of Hg, keeping 5 percent nitric acid (*v/v*) and 0.10 percent potassium dichromate (*m/v*) concentration. This standard solution is generally stable for a period of at least one month.

c) *Procedure* — Using the above standard solution, find out the transmission for 10, 20, 40, 60, 80, 100, 120, 140 and 150 nanogram of mercury. By following procedure prescribed in **L-3.2.3.3**, prepare the standard graph indicating Hg in ng against absorbance.

**L-3.2.3.3** Add 8 ml of 10 percent nitric acid and 2 ml of stannous chloride solution to the reaction vessel and stir the mixture for 5 min. Remove the blank transmission. Add 5 ml of prepared solution (*see* **L-3.4.1**). Volume in the reaction vessel should not be more than 15 ml in any case. Stir for 5 min and find out the transmission. This gives 'Back reading' (BR). Take the transmission for 5 ml of blank solution following same procedure. This gives 'Blank reading' (BLR).

Difference in the two readings (BR and BLR) corresponds to mercury content in the sample which can be obtained by referring the chart of absorbance versus mercury in nanograms.

**L-3.2.4** *Calculation*

$$\text{Mercury (as Hg), ppm by mass} = \frac{0.0004 \times X}{rd}$$

where

$X$ = mercury for transmission from the graph (BR - BLR), in nanograms; and

$rd$ = relative density (*see* Annex A).

### L-3.3 Mercury in Technical and Boiler Water Grades

**L-3.3.1** *Preparation of Sample Solution*

Take 5 ml of technical grade of the material (5 ml for boiler water grade also) and follow the procedure prescribed for the preparation of sample solution for pure grade acid as also for the blank solution.

**L-3.3.2** *Procedure*

Take 1 ml of sample solution (*see* **L-3.3.1**) and follow the procedure prescribed for pure grade acid (*see* **L-3.2.3.2** ).

**L-3.3.3** *Calculation*

$$\text{Mercury (as Hg ), ppm by mass} = \frac{0.02 \times X}{rd}$$

where

$X$ and $rd$ have the same significance as indicated in **L-3.2.4.**

# ANNEX M

[ *Table* 1, *Sl No.* (xi) ]

## METHOD OF TEST FOR TOTAL ORGANIC CARBON (TOC)

### M-1 OUTLINE OF THE METHOD

There are two common analysis methodologies known as differential and direct method. The differential method uses the technique where TOC is calculated by subtracting Total Inorganic Carbon (IC) from Total Carbon (TC) with the residual value being that of TOC. This method is commonly used in samples with less than or equal quantities or similar sized IC and TOC.

The second one being the direct method, which is used in cases where TOC is less than IC like-surface, ground and drinking water. In this method the sample is purged using an inert gas and TOC is estimated by oxidation. This process is also used for samples that might contain least or negligible quantities of TOC, which is also removed in the due course of this process. The basic principle in direct method is that, the measurement of $CO_2$ formed as a result of oxidation, gives the amount of TOC.

### M-2 APPARATUS

**M-2.1 Total Organic Carbon (TOC) Analyser**

### M-3 REAGENTS

**M-2.1 Potassium Hydrogen Phthalate —** AR Grade.

**M-2.2 Sodium Hydroxide (NaOH) AR grade**

**M-2.3 1 N Sodium Hydroxide Solution in TOC free water**

**M-2.4 Sodium Hydrogen Carbonate AR grade**

**M-2.5 Sodium Carbonate AR grade**

**M-2.6 Total Carbon (TC) Standard Solution** — Weigh accurately 2.125 gm potassium hydrogen phthalate that was previously dried at 120 °C for about 1 h and cooled in a desicator, transfer to 1 litre volumetric flask and make up to the mark with TOC free water. Label it as stock solution-1000 ppm TC. Dilute this solution 10 times with TOC free water to prepare 100 ppm TC solution.

**M-2.7 Inorganic Carbon (IC) Standard Solution** — Weigh accurately 3.5 gm of sodium hydrogen carbonate, which was previously dried for 2 h, at 280-290 °C and cooled in desiccator. Also weight out 4.41 g of sodium carbonate previously dried for 1 h at 280-290 °C and cooled in desiccator. Transfer the above weighed materials into one litre, volumetric flask and make up to the mark with zero water. It gives 1 000 ppm 'IC' standard solution prepare 100 ppm 'IC' solution by diluting 1 000 pm standard 'IC' solution ten times.

### M-3 PREPARATION OF IC AND TC CALIBRATION CURVES

Generate the calibration curves for IC and TC separately using respective standard solution. From "Calibration curve properties" windows, select the calibration points, that in. 10, 20, 50 and 100 ppm by auto dilution. First insert the TC solution (100 ppm) and run the programme. After completing the four standards insert the IC solution (100 ppm) and run the programme simultaneously and complete the four standard of IC solution. Use these two curves for sample analysis.

### M-4 PROCEDURE (DIFFERENTIAL METHOD)

Measure 25 ml of hydrochloric acid sample into a beaker, Add 25 ml of Millipore water or boiled and cooled distilled water. Add gradually 20 percent sodium hydroxide or 20 percent potassium hydroxide and reduce the PH to around 1.0 and thereafter add IN caustic slowly to adjust final pH between 2 and 3. Cool the solution, and make up to 500 ml. start the TOC analyzer. After furnace temperature reaches 680 °C, select 'TOC' method and the standard curve of TC and IC. Admit the sample in to the instrument and start the analysis. First the instrument reads the TC value and then the instrument reads IC value and automatic it gives the TOC value which is the difference of TC and IC values. Note the reading of the instrument (Final TOC value) and calculate the 'TOC' content of the sample.

### M-5 CALCULATION

TOC, in ppm = Instrument reading × 20.

# ANNEX N

( *Clause* 6.1 )

## SAMPLING OF HYDROCHLORIC ACID

### N-1 GENERAL REQUIREMENTS ON SAMPLING

**N-1.1** In drawing, preparing and storing test samples, the following precautions and directions shall be observed.

**N-1.2** Samples shall not be taken out in an exposed place.

**N-1.3** The sampling instrument shall be clean and dry.

**N-1.4** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**N-1.5** To draw representative sample, the contents of each container selected for sampling shall be mixed thoroughly by rolling, shaking or stirring by suitable means and with necessary caution.

**N-1.6** The samples shall be placed in suitable dry and airtight glass containers.

**N-1.7** Each sample container shall be sealed airtight after filling and shall be marked with full details of sampling, the data of sampling and the year of manufacture of the material.

### N-2 SCALE OF SAMPLING

**N-2.1 Lot**

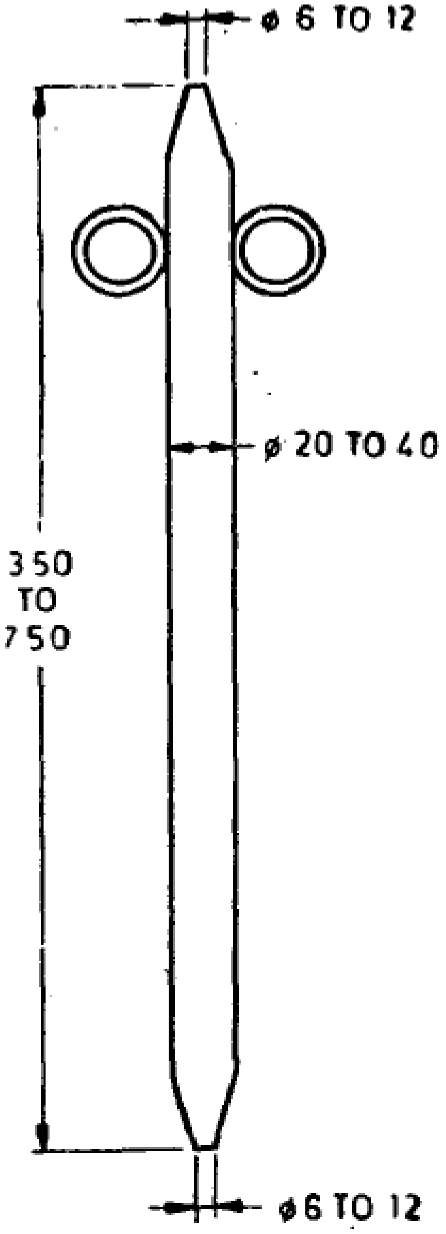
All containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**N-2.2** Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. For this purpose, five containers shall be selected at random from each lot.

> NOTE — If the number of containers in a lot is eight or less, the number of containers to be selected and the criteria for conformity of the lot to the specification requirements shall be as agreed to between the purchaser and the supplier.

**N-2.3** The containers shall be selected at random and to ensure randomness of selection, random number tables shall be used (*see* IS 4905). In case random number tables are not available, the following procedure may be used:

Starting from any container in the lot, count them as 1, 2, ......, up to $r$, and so on, where $r$ is the integral part of $N$ ($N$ being the number of containers in the lot). Every $r$th container thus counted shall be withdrawn to constitute a sample till the required number of five containers is obtained.



All dimensions in millimetres.

FIG. 3 SAMPLING TUBE

## N-3 PREPARATION OF TEST SAMPLES

### N-3.1 Sampling Tube

The sampling tube shall be made of glass and shall be 20 to 40 mm in diameter and 350 to 750 mm in length (*see* Fig. 3). The upper and lower ends are conical and reach 6 to 12 mm diameter at the narrow ends. For drawing the samples, the apparatus is first closed at the top with a thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material and is finally closed and withdrawn.

**N-3.1.1** For small containers, the size of the sampling tube may be altered suitably.

**N-3.2** For each of the containers selected according to **N-2.3** a small representative portion of the material, about 200 ml, shall be taken out with the help of the sampling tube after thoroughly stirring the acid with the help of a glass rod.

**N-3.3** Out of these portions, a small but equal quantity of the material shall be taken out and thoroughly mixed to form a composite sample, not less than 600 ml. The composite sample shall be divided into three equal parts, one for the purchaser another for the supplier and the third to be use as a referee sample.

**N-3.4** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the five containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**N-3.5** All individual composite sample shall be transferred to separate bottles. The bottles shall be sealed and labelled with full identification particulars.

**N-3.6** The referee samples consisting of composite sample and a set of five individual samples shall bear the seals of the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of a dispute between the two.

## N-4 NUMBER OF TEST

**N-4.1** Total acidity shall be tested on each of the five individual samples.

**N-4.2** Test for determination of remaining characteristics listed in Table 1 shall be performed on the composite sample.

## N-5 CRITERIA FOR CONFORMITY

**N-5.1** For individual samples for the five test results for total acidity, the mean X and range $R$ shall be computed (range being defined as difference between the maximum and minimum values of test results).

The lot shall be declared as conforming to the requirement of total acidity, if the value of the expression X-0.6 $R$ is greater than or equal to the relevant value specified in Table 1.

### N-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant value specified in Table 1.

**N-5.3** A lot shall be declared as conforming to the requirements of this specification' if **N-5.1** and **N-5.2** are satisfied.

# ANNEX P

( *Foreword* )

## COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

| *Organization* | *Representative(s)* |
|---|---|
| Central Salt and Marine Chemicals Research Institute, Bhavnagar | Dr Kannan Srinivasan (***Chairman***) |
| Aditya Birla Chemical (I) Ltd, New Delhi | Shri Alok Singh |
| Alkali Mfrs Association of India, Delhi | Shri K. Srinivasan<br>Shri Subhash Tandon (*Alternate*) |
| Bhabha Atomic Research Centre, Mumbai | Dr A. V. R. Reddy<br>Dr S. N. Achary (*Alternate*) |
| Central Drugs Standard Control Organization | Dr Raman Mohan Singh |
| DGQA, New Delhi | Dr Gurbachan Singh<br>Shri B. S. Tomar (*Alternate*) |
| Geological Survey of India, Kolkata | Dr D. K. Das<br>Dr Subhas Chandra (*Alternate*) |
| Grasim Industries Ltd, Nagda | Shri R. S. Baghel<br>Shri Pankaj Gupta (*Alternate*) |
| Gujarat Alkalies and Chemicals Ltd, Vadodara | Dr Sunil Sinha |
| Hindustan Lever Ltd, Mumbai | Vrinda Rajwade<br>Shrimati Poornakala (*Alternate* I)<br>Satyamoorthy (*Alternate* II) |
| In personal capacity | Dr A. N. Bhat |
| In personal capacity | Dr T. S. Kathpal |
| Industrial Carbon Pvt Ltd, Ankleshwar | Shri Rohit Kumar Madhavji<br>Shri Satyan Rohit Kumar (*Alternate*) |
| Indian Institute of Chemical Technology, Hyderabad | Dr Praveen R. Likhar<br>Dr Rajender Reddy (*Alternate*) |
| Ministry of Defence (DGQA), Kanpur | Shri R. N. Aparajit |
| Ministry of Chemicals & Fertilizers | Dr Rohit Misra |
| National Chemical Laboratory, Pune | Dr Darbha Srinivas<br>Dr Paresh Dhepe (*Alternate*) |
| National Metallurgical Laboratory, Jamshedpur | Dr Trilochan Mishra<br>Shri Devbrata Mishra (*Alternate*) |
| National Mineral Development Corporation Ltd., Hyderabad | Shri Rajan Kumar<br>Dr Prashant Sharma (*Alternate*) |
| National Physical Laboratory, New Delhi | Dr Nahar Singh<br>Dr S. P. Singh (*Alternate*) |
| National Test House (NR) | Dr Y. C. Nijhawan<br>Dr (Ms) A. Biswas (*Alternate*) |
| Nirma Chemicals, Ahmedabad | Shri R. A. Joshi<br>Dr K. C. Pathak (*Alternate*) |

| *Organization* | *Representative(s)* |
|---|---|
| Office of the Development Commissioner (MSME), New Delhi | Shrimati Shimla Meena<br>Shri Santosh Kumar (*Alternate*) |
| Reliance Industries Ltd, Navi Mumbai | Shri Dhanavadan Modi |
| Shriram Institute for industrial research, Delhi | Dr (Ms) Laxmi Rawat<br>Shri B. Govindan (*Alternate*) |
| VOICE, Delhi | Shri H. Wadhwa<br>Shri K. C. Chaudhary (*Alternate*) |
| In Personal Capacity | Shri D. K Jain |
| Vaibhav Analytical Services, Ahmedabad | Shri Gaurang Oza |
| BIS Directorate General | Shri A. K. Lal, Scientist 'E' and Head (CHD)<br>[ Representing Director General ( *Ex-officio* ) ] |

*Member Secretary*

Sagar Singh
Scientist 'C' (CHD), BIS

**Bureau of Indian Standards**

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

**Copyright**

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

**Review of Indian Standards**

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 01 (11222).

## Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
| | | |
| | | |
| | | |

**BUREAU OF INDIAN STANDARDS**

**Headquarters:**

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
*Telephones*: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

**Regional Offices:** *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi
KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg
CHANDIGARH 160019 — 265 0206, 265 0290

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

**Branches** : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi